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DRAWINGS ATTACHED

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(54) A STABLE DISPERSION OF SYNTHETIC DIAMOND POWDER

(71) We, ALLIED CHEMICAL CORPORATION, a Corporation organised and existing under the laws of the State of New York, United States of America of, 61 Broadway, New York 6, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to novel stable diamond dispersions and to the method of preparing them.

Synthetic shock-formed diamonds are known. Such diamonds can be obtained, for example, by processes such as that described in our U.S. Patent No. 3,238,019. The diamonds made by that process are characterised by being a powder composed of individual diamond particles having an average diameter in the range from 7×10^{-4} to 1×10^{-2} microns and having a surface area between 40 and 400 square metres per gram. At least 10% of the surface area contains hydroxyl, carboxyl and carbonyl functional groups. The diamond powder is contaminated with agglomerates of graphite, siliceous matter and particles of diamond strongly agglomerated together, the agglomerates having an average diameter within the range 1-5 microns. The synthetic diamond powder is more particularly described in our Patent Specification No. 1,154,633. Although the synthetic diamond powder of the above-identified prior application is preferred, any shock-formed synthetic diamonds within the specified particle size range can be used in the process of the present invention. Synthetic diamonds tend to be obtained as large aggregates of fine particles.

Examples of uses of such a synthetic shock-formed diamond powder are as an abradant in lapping compositions and as a wear resistant element incorporated in metal and plastic surfaces.

For both of these uses and for many other uses it is highly desirable to disperse the diamond powder uniformly in a liquid dispersion medium so as to form a stable dispersion. Such a dispersion can be used directly as a lapping compound or can be employed as an intermediate stage in the formation of materials having wear resistant surfaces. For example, the dispersion can be formed in a polymerisable liquid which is then polymerised to form a solid so as to incorporate the diamond powder therein.

For these and many other uses it is necessary to break up the large diamond aggregrates into their smaller component parts or to separate the aggregates from the smaller particles. This is necessary since for many applications the aggregates are too coarse to serve as an abradant and when used result in a scratched and unacceptable surface.

Many different techniques were tried in order to form stable synthetic diamond dispersions having particles of uniform size range, i.e. in which the aggregates have been broken up. However, it was not found possible by known techniques to segregate synthetic diamonds on a large scale or in high concentrations, or to break up the aggregates so as to make segregation unnecessary. Furthermore, it was found that there was no known way to make a stable dispersion in which the concentration of the synthetic diamonds equalled or exceeded 0.5% by weight. (Here and throughout this specification we refer to concentrations of diamonds as percentages by weight of the liquid dispersion medium.) For example, addition of peptisable colloids like gelatin, gum arabic or soap did not prevent flocculation after short standings of even a 1% by weight dispersion. It was found that stable dispersions of useful quantities of diamond powder in, for example, olive oil, water, glycol or Varsol ("Varsol" is a Registered Trade Mark) could not be formed by conventional methods such as hand or automatic mortars, ball mills or even ultrasonic waves. When prepared by such means the dispersions showed poor qualities of stability even at low diamond concentrations. For example, no known

dispersion containing 0.5% or more by weight of diamonds was stable over about 12 hours. Furthermore, it was found that large agglomerates remained in the dispersions even, for example, after exposing the diamond material to a ball mill for a long period of time.

It is an object of this invention to provide stable diamond dispersions containing from 0.5 to 15% diamond powder, by weight of dispersion medium, in a liquid dispersion medium.

We have now found that stable dispersions of from 0.5 to 15% by weight of synthetic shock-formed diamond powder can be obtained by subjecting a mixture of synthetic diamond powder of the average particle size described above in a liquid dispersion medium to the high shear stresses that can be obtained with a rotating blade mixer rather than with, e.g., a ball mill.

The invention provides a method of preparing a stable dispersion, in a liquid dispersion medium, of synthetic shock-formed diamond powder, whose particles have an average diameter of 7×10^{-4} to 1×10^{-2} microns and remain dispersed in the medium for more than 24 hours, the method comprising dispersing in the medium, by means of rotating blades, a charge comprising 0.5 to 15% by weight on the weight of the medium, of the synthetic diamond powder, a surfactant being also present if the surface tension of the medium is less than 35 dynes/cm, and, when the medium is water, the medium having a pH of 5 to 10.2.

Preferably there are applied shear stresses of at least 0.83 watt per cc. for a period of at least two minutes. The preferred dispersions of this invention contain from 3 to 8% by weight of diamond powder. The liquid dispersion medium generally has a viscosity between 1/2 centipoise to 100 centipoises, preferably from $^1/_2$ to 5 centipoises. Dispersions can be formed in more viscous materials if upon heating the materials can be brought within the specified viscosity range. The liquid dispersion medium should have a surface tension above 35 dynes per centimetre and preferably between 45 and 85 dynes per centimetre. However, as indicated above, liquids having surface rensions below 35 dynes per centimetre can be used if a surfactant is employed therewith.

Suitable dispersion media include vegetable oils such as olive oil, polyols such as glycol, and commercially available solvent mixtures such as Varsol.

The amount of diamond powder added to the liquid dispersion medium before exposing the mixture to the shear stresses is from $^1/_2$ gram to 15 grams per 100 g. of liquid, preferably from 3 grams to 8 grams per 100 g.

The temperature at which the shear stresses are applied is not critical as long as it is sufficient to maintain the dispersion medium in the liquid phase and within the above viscosity range. However, a temperature of 40°C or above is preferred.

The shear stresses can be applied to the mixture of diamonds in liquid dispersion medium in a variety of ways. For example conventional homogenizers or high-speed commercial or household blenders can be used. The latter is preferred because of its inexpensiveness and ready availability. However, it should be noted that this listing is merely exemplary and not intended to be limiting and that any rotating blade equipment, which high shear stresses, can also be used.

It is surprising that whereas a ball mill does not form a dispersion of synthetic diamond which is stable for 24 hours, one can prepare such a dispersion with a rotating blade mixer.

The energy supplied to the diamond-liquid mixture in the form of shear stresses is desirably in the range from 250 to 1000 watts, preferably between 400 and 500 watts, for a charge of diamond containing liquid of a volume from 50 to 300 cc., i.e. a minimum of 0.83 watt per cc. This energy should for best results be applied to the charge at a speed between about 8,000 and 20,000 r.p.m., preferably between 15,000 and 19,000 r.p.m., over a period from 2 to 60 minutes, preferably from 10 to 20 minutes.

Applicants have found that the dispersions of this invention can be prepared in any liquid medium having the viscosity and surface tension characteristics outlined above. Examples of such media are Varsol, ethanol, olive oil, glycol, glycerine, hydrocarbons halogenated hydrocarbons such as 1,1,2 - trichloro - 1,2,2 - trifluoroethane, water and mixtures thereof. However, it has been found that when water is used as the dispersion medium the medium must satisfy another requirement in order to successfully disperse synthetic shock-formed diamond powder, i.e. the pH must be maintained between 5 and 10.2, preferably between 7 and 8.5. As disclosed above, when the surface tension of the suspending medium used is below 35 dynes per cm., a conventional surfactant should be used in order to obtain stable dispersions. Such a surfactant should be added in the amount of from 0 to 15% by weight, based on the weight of the dispersion medium preferably from 2 to 4% by weight. Examples of such surfactants are alkyl aryl sulphonates such as EMCOL P-10-59 and EMCOL P-5900 ("Encol" is a Registered Trade Mark; products of Witco Chemical Co., Inc.). Other conventional surfactants may also be employed.

The following is a description of a technique which is exemplary of the pre-

ferred embodiment of the novel method of this invention.

A 200 to 210 cc. charge of olive oil or other suitable dispersion medium is introduced into the removable jar of a household blender having a 1 litre capacity bowl, 4-one inch long blades and a variable speed 1/4 HP motor. 0.08 to 0.10 grams of synthetic diamond powder, made by the process of U.S. Patent 3,238,019, per cc. of charge is added to the olive oil and the blender is switched on and run for 10 to 20 minutes at 15,000 to 19,000 r.p.m. The resultant dispersion in olive oil is stable for about a week.

By stability applicant means that the diamonds remain dispersed in the liquid. For the purposes of this application dispersion means the condition in which the diamond particles are each surrounded by molecules of dispersion medium and, for the most part, do not touch each other. Although the dispersions of this invention may exhibit some degree of phase separation or settling after prolonged periods of standing, such as several days, such settling does not amount to sedimentation and the diamond powder remains dispersed within the meaning of the above definition. Such settled dispersions are easily restored to their original condition by simple stirring or shaking and then do not exhibit settling again for prolonged periods, i.e. more than 24 hours.

Dispersions of diamond powder, from the process of U.S. Patent 3,238,019, were prepared according to substantially the above procedure using glycol, water of pH 10.1 and Varsol plus about 4.0% by weight surfactant (EMCOL P-10-59. All of these dispersions were found to be stable for over 24 hours.

The accompanying drawings are photomicrographs which compare applicant's novel dispersions with dispersions made by prior art techniques. The scale is indicated on each figure. The drawings are as follows:

Figure 1 is a photomicrograph of synthetic diamonds dispersed in olive oil according to the present invention, the diamonds being present in 0.5% concentration by weight. This figure shows the pronounced effect of shearing stresses in breaking up agglomerates and ensuring a uniform dispersion. The diamonds were dispersed by applicants' above-described technique.

Figure 2 is a photomicrograph of dispersed synthetic diamonds in olive oil, 0.1% of diamonds by weight of oil. This figure shows the agglomerates which were still present after 8 hours of operation on the dispersion of a Spex Mixer Mill, a type of ball mill.

Figure 3 is a photomicrograph of an 8% concentration of synthetic diamonds dispersed in olive oil by the use of the above-described technique according to the invention.

Figures 4 and 5 are photomicrographs of synthetic diamonds dispersed in water having a pH of 10.1 by the technique of this invention. Figure 4 shows a 0.5% concentration by weight and Figure 5 shows an 8% concentration by weight.

Figures 6 and 7 are photomicrographs of synthetic diamonds dispersed in Varsol by the technique of this invention using a surfactant, EMCOL P-10-59. Figure 6 shows a 0.5% concentration and Figure 7 shows an 8% concentration.

Figure 8 is a photomicrograph of settled but still dispersed synthetic diamonds in Varsol at 0.5% concentration. The dispersion was prepared by the technique of this invention and then allowed to stand for about 16 hours.

Figure 9 is a photomicrograph of the same synthetic diamonds as shown in Figure 8 after redispersion by simple shaking.

Figure 10 is a photomicrograph for comparison of a 0.1% concentration of dispersed synthetic diamonds in water at pH 10.1. The agglomerates which are still present after 6 hours of Spex Mixer Mill operation on the dispersion are apparent.

Figure 11, which should be contrasted with Figure 10, is a photomicrograph of dispersed synthetic diamonds in water at pH 10.1, 0.5% concentration, prepared by the technique of this invention. This figure shows the pronounced effect of shearing stresses in breaking up agglomerates which are not broken up by other methods.

It is evident from the drawings that by use of a rotating blade mixer which gives rise to higher shear stresses than a ball mill a surprisingly better dispersion is achieved than with a ball mill.

WHAT WE CLAIM IS:—

1. A method of preparing a stable dispersion, in a liquid dispersion medium, of synthetic shock-formed diamond powder, whose particles have an average diameter of 7×10^{-4} to 1×10^{-2} microns and remain dispersed in the medium for more than 24 hours, the method comprising dispersing in the medium, by means of rotating blades, a charge comprising 0.5 to 15% by weight, based on the weight of the medium, of the synthetic diamond powder, a surfactant being also present if the surface tension of the medium is less than 35 dynes/cm, and, when the medium is water, the medium having a pH of 5 to 10.2.

2. A method according to claim 1 wherein the powder is dispersed by a homogenizer.

3. A method according to claim 1 wherein the powder is dispersed by a blender.

4. A method according to claim 1 wherein said blades operate at from 8000 to 20,000 r.p.m.

5. A method according to any one of the

preceding claims in which the charge is subjected to at least 0.83 watt of shear stress per cc. of dispersion medium.

6. A method according to claim 5 wherein the shear stress applied is from 250 to 1000 watts per charge of 50-300 cc.

7. A method according to claim 6 wherein the shear stress is from 400 to 500 watts per charge of 50-300 cc.

8. A method according to any one of the preceding claims wherein the dispersion medium is a vegetable oil.

9. A method according to any one of claims 1 to 7 wherein the dispersion medium is a polyol.

10. A method according to any one of the preceding claims wherein the dispersion medium has a viscosity at the mixing temperarure between $^1/_2$ centipoise and 100 centipoises.

11. A method according to any one of the preceding claims wherein the synthetic diamond powder has an average particle diameter of 7×10^{-4} to 1×10^{-2} micron, a surface area of 40 to 400 square metres per gram and at least 10% of the surface area contains hydroxyl, carboxyl and carbonyl functional groups.

12. A method according to any one of the preceding claims wherein the shear stresses are applied over a period of 2 to 60 minutes.

13. A method according to claim 12 wherein the period is 10 to 20 minutes.

14. A method according to claim 1 of preparing a stable dispersion of synthetic shock-formed diamond in a liquid dispersion medium, substantially as hereinbefore described.

15. A dispersion of synthetic diamond prepared by a method according to any one of the preceding claims.

16. A dispersion of 0.5 to 15% by weight of shock-formed synthetic diamond powder, whose particles have an average diameter of 7×10^{-4} to 1×10^{-2} microns, in a liquid dispersion medium, said medium having a viscosity of from $^1/_2$ centipoise to 100 centipoises, said dispersion being stable for more than 24 hours, a surfactant being present if the surface tension of the medium is less than 35 dynes/cm, and, when the medium is water, the medium having a pH of 5 to 10.2.

17. A dispersion according to claim 16 wherein said synthetic diamond powder has a surface area of 40 to 400 square metres per gram and at least 10% of the surface area contains hydroxyl, carboxyl and carbonyl functional groups.

18. A dispersion according to claim 16 or 17 wherein the dispersion medium is olive oil and which is stable for about one week.

19. A dispersion according to claim 16 or 17 wherein the dispersion medium is glycol.

20. A dispersion according to any one of claims 16 to 19 wherein the synthetic diamond powder is present in an amount from 3 to 8% by weight of the dispersion medium.

21. A stable dispersion of synthetic diamond according to claim 16 and substantially as hereinbefore described.

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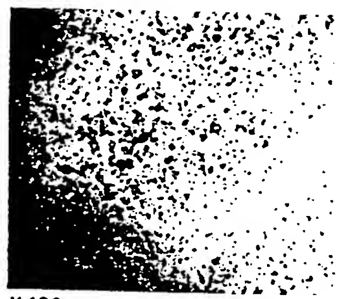
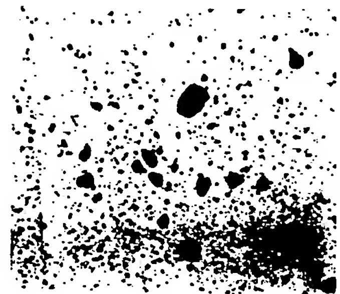
1210299 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1



X420 FIG. 1.



X132 FIG. 2.

X420 FIG. 3.

X420 FIG. 4.

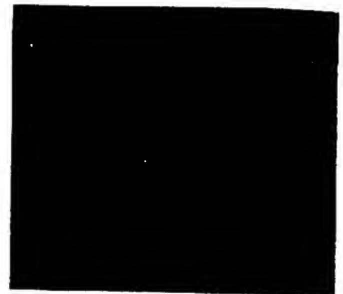
X420 FIG. 5.

1210299 COMPLETE SPECIFICATION

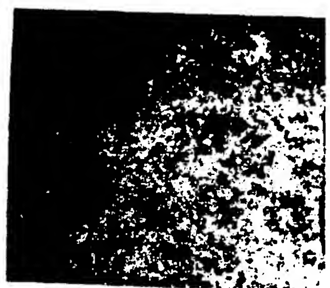
2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

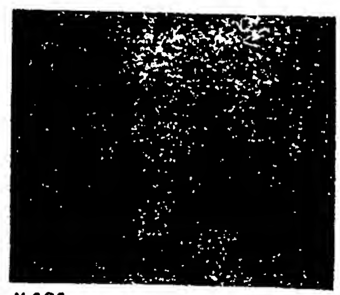
X 420 FIG. 6.



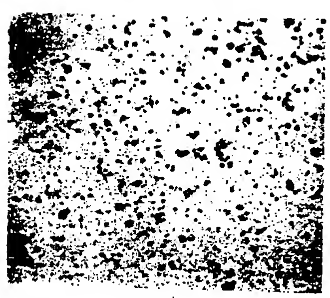
X 420 FIG. 7.



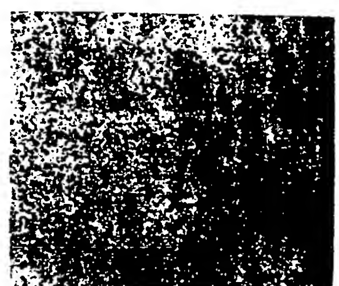
X 420 FIG. 8.



X 420 FIG. 9.



X 300 FIG. 10.



X 420 FIG. 11.